

Antiferromagnetic Order and Superconductivity in $\text{Sr}_4(\text{Mg}_{0.5-x}\text{Ti}_{0.5+x})_2\text{O}_6\text{Fe}_2\text{As}_2$ with Electron Doping: ^{75}As -NMR Study

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(Received February 27, 2013)

We report an ^{75}As -NMR study on iron (Fe)-based superconductors with thick perovskite-type blocking layers $\text{Sr}_4(\text{Mg}_{0.5-x}\text{Ti}_{0.5+x})_2\text{O}_6\text{Fe}_2\text{As}_2$ with $x=0$ and 0.2. We have found that antiferromagnetic (AFM) order takes place when $x=0$, and superconductivity (SC) emerges below $T_c=36$ K when $x=0.2$. These results reveal that the Fe-pnictides with thick perovskite-type blocks also undergo an evolution from the AFM order to the SC by doping electron carriers into FeAs planes through the chemical substitution of Ti^{+4} ions for Mg^{+2} ions, analogous to the F-substitution in LaFeAsO compound. The reason why the $T_c=36$ K when $x=0.2$ being higher than the optimally electron-doped LaFeAsO with $T_c=27$ K relates to the fact that the local tetrahedron structure of FeAs_4 is optimized for the onset of SC.

KEYWORDS: $\text{Sr}_4(\text{Mg,Ti})_2\text{O}_6\text{Fe}_2\text{As}_2$, perovskite blocks, superconductivity, antiferromagnetism, NMR

Iron-based high- T_c superconductors comprise a two-dimensional layered structure of iron (Fe)-pnictogen (Pn) planes,¹ which are separated by blocking layers, such as LnO (Ln =rare earth), alkaline earth metals and alkaline metals, and so on. Relatively high superconducting transition has been reported in Fe-pnictides with thick perovskite-type blocking layers, for example, T_c is ~ 47 K for $\text{Ca}_4(\text{Mg}_{0.25}\text{Ti}_{0.75})_3\text{O}_y\text{Fe}_2\text{As}_2$ ² and ~ 37 K for $\text{Sr}_4\text{V}_2\text{O}_6\text{Fe}_2\text{As}_2(\text{V}-42622)$,³ in which an interlayer distance between FePn layers (L) is longer than 13 \AA .²⁻⁵ However, a ground state of undoped $(\text{FeAs})^-$ layer and a relation with the onset of SC in the Fe-pnictides with thick perovskite blocks has not been identified sufficiently, although it is well known that the superconductivity (SC) in Fe-pnictide compounds emerges in close proximity to antiferromagnetism (AFM).¹ In $\text{Sr}_4(\text{Mg}_{0.5-x}\text{Ti}_{0.5+x})_2\text{O}_6\text{Fe}_2\text{As}_2$ (denoted as $\text{Mg}_{0.5-x}\text{Ti}_{0.5+x}$ -42622 hereafter), it has been reported that T_c increases from 0 K to 36 K by the substitution of Ti for Mg,⁴ and up to 43 K by the application of high pressure.⁷ In this compound with $x=0$, the Fe^{2+} state is formally expected as well as other parent Fe-pnictide compounds.¹ Therefore, systematic investigations of these compounds will provide us with further insight into the intimate relationship between SC and AFM order inherent in FeAs layers with a highly two-dimensional electronic structure.

In this Letter, we report ^{75}As -nuclear magnetic resonance (NMR) study of $\text{Mg}_{0.5-x}\text{Ti}_{0.5+x}$ -42622 with $x=0$ and 0.2, which unravels that an AFM order sets in at $x=0$ and an SC state emerges at $x=0.2$ by doping electron carriers into the FeAs layers through the chemical substitution of Ti for Mg. We remark that the ground state of $(\text{FeAs})^-$ layer of undoped $\text{Mg}_{0.5-x}\text{Ti}_{0.5+x}$ -42622 resembles those of 1111 and 122 systems.

Polycrystalline samples of $\text{Mg}_{0.5-x}\text{Ti}_{0.5+x}$ -42622 were synthesized in quartz ampules at ambient pressure as described elsewhere.⁴ Powder x-ray diffraction measurement indicates that the samples are dominated by an intrinsic phase, whereas the $x=0.2$ sample contains small amounts of impurities such as SrFe_2As_2 and Sr_2TiO_4 .⁴ However, the ^{75}As -NMR signal inherent in $\text{Mg}_{0.5-x}\text{Ti}_{0.5+x}$ -42622 at $x=0.2$ is discriminated from that of SrFe_2As_2 .⁸ Both the samples with $x=0.0$ and 0.2 are the same lattice parameters: a - and c -axis length of $a=3.94 \text{ \AA}$ and $c=15.95 \text{ \AA}$, a height of pnictogen from Fe-plane $h_{Pn} \sim 1.4 \text{ \AA}$, and a Pn -Fe- Pn bond angle, $\alpha \sim 109.5^\circ$.² These parameters are comparable to the optimum lattice parameters to reach a highest T_c in various series of Fe based compounds, as suggested in the literature.^{9,10} The T_c s of $\text{Mg}_{0.5-x}\text{Ti}_{0.5+x}$ -42622 determined by the resistivity and susceptibility measurements⁴ are shown in Fig. 1(a). Here, $T_{c(\rho)}^{\text{onset}}$ and $T_{c(\rho)}^{\text{zero}}$ are the respective temperatures for an onset and zero resistance of SC in resistivity measurement, and $T_{c(\chi)}^{\text{onset}}$ is a temperature for an onset of SC diamagnetism in susceptibility measurement. Note that $T_{c(\rho)}^{\text{zero}} \sim 5$ K when $x=0$ is not associated with a bulk SC, but a filamentary-induced one, since the SC diamagnetism does not point to a bulk nature.⁴ The substitution of Ti for Mg brings about a bulk SC transition at $T_{c(\rho)}^{\text{onset}} \sim 36$ K ($T_{c(\rho)}^{\text{zero}} \sim 22$ K), causing the distinct appearance of SC diamagnetism. ^{75}As -NMR measurements have been performed for coarse powder samples of $\text{Mg}_{0.5-x}\text{Ti}_{0.5+x}$ -42622 ($x=0$ and 0.2).

Figure 1(c) shows the temperature(T)-dependence of ^{75}As -NMR ($I=3/2$) spectra for the powder sample of $\text{Mg}_{0.5}\text{Ti}_{0.5}$ -42622 with $x=0$. The spectrum at 100 K is a typical powder pattern affected by the nuclear quadrupole interaction in a paramagnetic state. Here, $^{75}\nu_Q$ was estimated to be ~ 11.7 MHz, which is slightly larger than in LaFeAsO .¹¹ As T lowers, the spectrum

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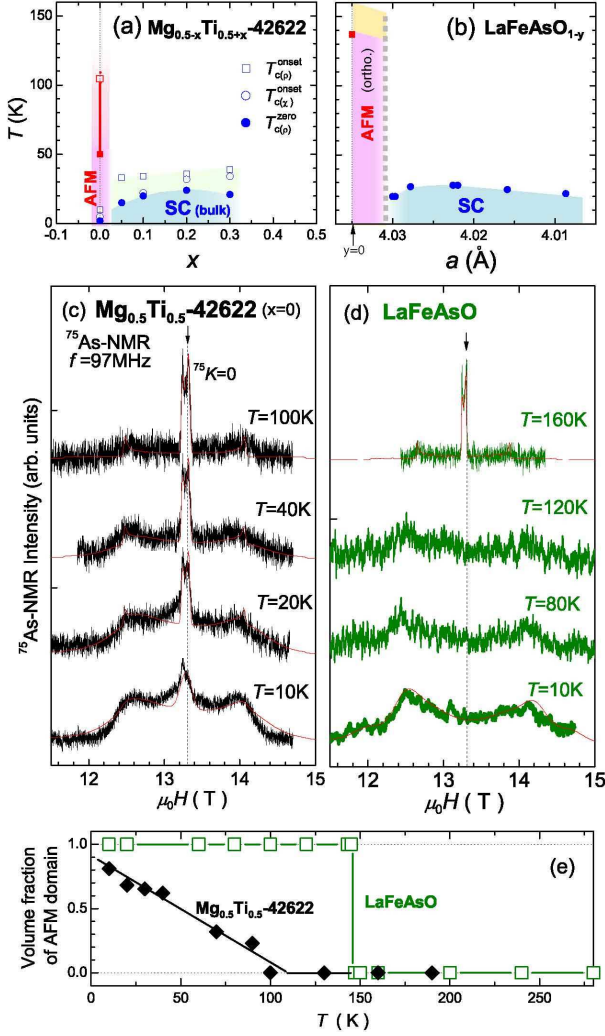


Fig. 1. (Color online) (a) Phase diagram of $\text{Mg}_{0.5-x}\text{Ti}_{0.5+x}-42622$. Respective $T_{\text{onset}}^{c(\rho)}$ and $T_{\text{onset}}^{c(\chi)}$ are the onset of resistivity drop and SC diamagnetism in susceptibility, and $T_{\text{zero}}^{c(\rho)}$ presents zero resistivity [Sato et al.⁴]. The Néel temperature (T_N) for $x=0$ is 50 ~ 100 K, as revealed in this study (see text). (b) Phase diagram of LaFeAsO_{1-y} .¹² (c) and (d) are T -dependence of ^{75}As -NMR spectra for $x=0$ and LaFeAsO ($T_N=140$ K), respectively. The solid lines are the simulated spectra when the internal field $H_{\text{int}}^{\parallel c}$ and their ν_Q values at the As site are assumed in these compounds. (e) T dependence of volume fraction of AFM domain evaluated from the fractional intensity of the broad spectra. Here, we assume that the difference of spin-spin relaxation time T_2 in two phases is neglected.

overlaps with a broad spectrum with its spectral intensity being large below 100 K. Note that the broad NMR spectral shape resembles that of the parent compound LaFeAsO , which exhibits the commensurate stripe AFM order below $T_N \sim 140$ K. Accordingly, the spectra for $\text{Mg}_{0.5}\text{Ti}_{0.5}-42622$ below 100 K are composed of an AFM ordered phase and a paramagnetic one, which is evidence of phase separation in the sample. As shown in Fig. 1(e), the volume fraction of the AFM domain evaluated from the fraction of the broad spectra increases at low temperatures, suggesting that the AFM domain size may develop spatially upon cooling in association with a possible inhomogeneity of the local concentration of Mg/Ti atoms. As shown in the solid curve in Fig. 1(c),

Table I. Internal field at respective ^{57}Fe and ^{75}As sites from ^{57}Fe Mössbauer and ^{75}As -NMR studies for the undoped Fe-pnictides at low temperatures, together with the ordered moment (M_{AFM}) and T_N .¹⁴

	$^{57}H_{\text{int}}[\text{T}]$ (^{57}Fe site)	$^{75}H_{\text{int}}[\text{T}]$ (^{75}As site)	M_{AFM}^{14}	$T_N[\text{K}]^{14}$
$\text{Mg}_{0.5}\text{Ti}_{0.5}-42622(x=0)$		$\pm 1.3^\dagger$		50~100 [†]
LaFeAsO	5.3 ¹⁵	$\pm 1.6^{12}$	$0.8\mu_B^{16}$	140
BaFe_2As_2	5.47 ¹⁷	$\pm 1.4^{8,20}$	$0.87\mu_B$	140
SrFe_2As_2	8.9 ¹⁸	$\pm 2.2^8$	$1.01\mu_B$	220
CaFe_2As_2	10 ¹⁹	$\pm 2.6^{21}$	$0.8\mu_B$	173
$\text{Sc}-42622$	1.65 ⁶		$0.11\mu_B^6$	50 ⁶

†) results on this experiment

the broad spectra of AFM domains are tentatively reproduced by assuming $^{75}H_{\text{int}}^{\parallel c} \sim \pm 1.3$ T and $^{75}\nu_Q^{\parallel c} = 11.7$ MHz. On the other hand, it should be noted that the ^{75}As NMR spectrum of LaFeAsO is well reproduced by assuming an internal field $^{75}H_{\text{int}}^{\parallel c} = \pm 1.6$ T and $^{75}\nu_Q^{\parallel c} = 8.8$ MHz, revealing that no phase separation takes place in LaFeAsO , as indicated by the solid line in Fig. 1(d).¹² Table I presents a list of the internal fields at ^{57}Fe and ^{75}As sites, T_N and ordered moments M_{AFM} derived from the experiments of ^{57}Fe -Mössbauer and ^{75}As -NMR on mother compounds of FeAs based superconductors. The $^{57}H_{\text{int}}$ and $^{75}H_{\text{int}}$ are induced by M_{AFM} through the respective hyperfine-coupling constants $^{57}A_{hf}$ and $^{75}B_{hf}$. In particular, the origin of $^{75}B_{hf}$ is attributed to an off-diagonal pseudodipole field induced by stripe-type AFM ordered moments lying on the ab-plane at the Fe site.¹³ Since $^{75}H_{\text{int}}^{\parallel c} \sim \pm 1.3$ T in $\text{Mg}_{0.5}\text{Ti}_{0.5}-42622$ is comparable with those values in LaFeAsO and BaFe_2As_2 , it is likely that its AFM ordered state is similar to those in LaFeAsO and BaFe_2As_2 . It differs from the static magnetic order of tiny moment from the FeAs layer, which was reported in $\text{Sr}_4\text{Sc}_2\text{O}_6\text{Fe}_2\text{As}_2$ (denoted as $\text{Sc}-42622$).⁶ Hence, we remark that a commensurate stripe AFM order being comparable to LaFeAsO and BaFe_2As_2 is realized in the ground state of $\text{Mg}_{0.5}\text{Ti}_{0.5}-42622$, even though Mössbauer and neutron scattering experiments in this compound are not yet reported.

In the powder sample of $\text{Mg}_{0.3}\text{Ti}_{0.7}-42622$ with $x=0.2$, the broad spectrum arising from the AFM domains was not observed, suggesting that doping electron carriers expel the AFM domains. This fact suggests that Ti ions are in a tetravalent state of Ti^{4+} with $3d^0$ in blocking layers, which contrasts with the trivalent state of V^{3+} ions in $\text{Sr}_4\text{V}_2\text{O}_6\text{Fe}_2\text{As}_2$ which are magnetic.^{7,22,23} Thus, the substitution of nonmagnetic Ti^{4+} ions for Mg^{2+} ions results in an increase in electron density and leads to the collapse of the AFM order. This is also corroborated by the fact that $^{75}\nu_Q \sim 12.6$ MHz at $x=0.2$ is slightly larger than that at $x=0$, which also resembles the doping dependence of $^{75}\nu_Q$ in LaFeAsO system.^{11,24}

The nuclear spin-lattice relaxation rate $1/T_1$ was measured at the central peak in the ^{75}As -NMR spectra (see Fig. 1(c)). Here, $1/T_1$ was determined from the recovery curve of ^{75}As nuclear magnetization following the theo-

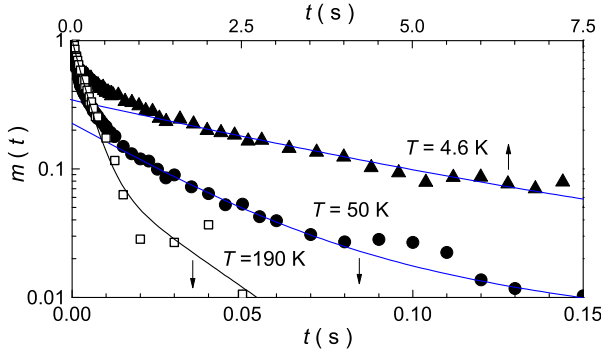


Fig. 2. (Color online) Recovery curves of ^{75}As nuclear magnetization in $x=0$. The $1/T_1$ can be determined by a single theoretical curve at high temperatures ($T > 120$ K), but not below 100 K. The fraction of the long T_1 component becomes large at low temperatures in accordance with the emergence of broad spectra below 100 K (See Fig. 1(c)).

retical function for $I = 3/2$: $m(t) = [M_0 - M(t)]/M_0 = 0.1 \exp(-t/T_1) + 0.9 \exp(-6t/T_1)$, where M_0 and $M(t)$ are the respective nuclear magnetizations for the thermal equilibrium condition and at a time t after the saturation pulse. As shown in Fig. 2, the $m(t)$ at $x=0$ is fitted by a theoretical curve with a single component of T_1 at temperatures higher than 120 K, but not lower than ~ 100 K. Therefore, a long component T_{1L} and a short component T_{1S} are tentatively deduced by assuming an expression given by $m(t) \equiv A_S m_S(t) + A_L m_L(t)$. Here, A_S and A_L with $A_S + A_L = 1$ represent the respective volume fractions of domains with T_{1S} and T_{1L} . Note that A_L becomes larger upon cooling below ~ 100 K in association with the emergence of AFM domains. Accordingly, it would be expected that T_{1L} and T_{1S} are associated with the AFM domains and the paramagnetic domains, respectively, reflecting the phase separation in $\text{Mg}_{0.5}\text{Ti}_{0.5}\text{-42622}$ with $x=0$.

The T dependences of $1/T_{1S}T$ and $1/T_{1L}T$ components are plotted in Fig. 3(a). The $1/T_{1S}T$ increases upon cooling below 100 K, but it decreases rapidly with a peak at 50 K, accompanied by a reduction in the volume fraction of the paramagnetic domains. On the other hand, $1/T_{1L}T$ decreases gradually upon cooling below ~ 100 K, accompanied by an increase in the volume fraction of the AFM domains. These results are consistently interpreted by the fact that AFM ordered domains develop progressively below ~ 100 K and their fraction exceeds the fraction of paramagnetic domains below 50 K, as presented in Fig. 1(e). The peak in $1/T_{1S}T$ at $T_N^* = 50$ K may suggest that some paramagnetic domains undergo an AFM order with a possible distribution of Néel temperature (T_N) in between 50 K and 100 K, depending on a possible spatial inhomogeneity of the local concentration of Mg/Ti atoms. It contrasts with the case of stoichiometric parent compounds LaFeAsO and BaFe_2As_2 , as compared in Fig. 1(e), where the Fe^{2+} states of LaFeAsO and BaFe_2As_2 are homogeneously realized on the $(\text{FeAs})^-$ layer without any phase separation after the structural transition to the orthorhombic phase. We also note that T_N s of $x=0$ and Sc-42622 in the previous report⁶ are sig-

nificantly lower than $T_N \sim 140$ K for other parent compounds LaFeAsO and BaFe_2As_2 , which may relate with the large interlayer distance between the FeAs layers.

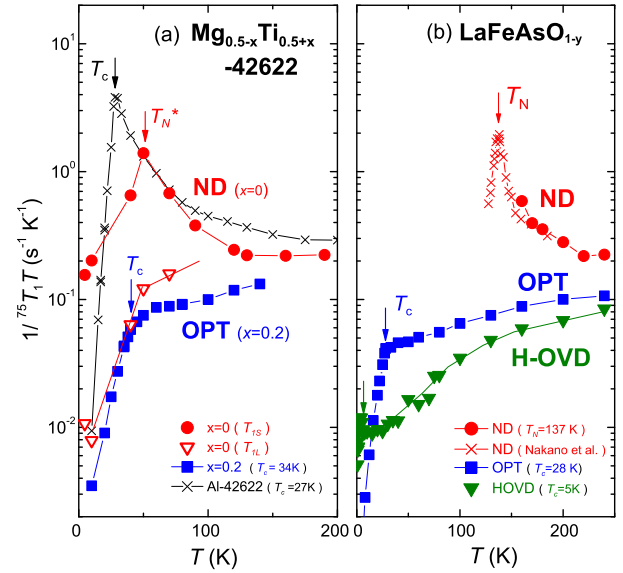


Fig. 3. (Color online) T dependence of ^{75}As -NMR- $(1/T_1T)$ for (a) $\text{Mg}_{0.5-x}\text{Ti}_{0.5+x}\text{-42622}$ ($x=0$ and 0.2) and Al-42622 ($T_c = 27$ K)²⁸, and (b) LaFeAsO system (non-doped (ND),^{12, 25} optimally-doped (OPT) with $T_c = 28$ K,¹¹ and heavily overdoped (H-OVD) with $T_c = 5$ K²⁶).

Next, we deal with the T_1 results for $\text{Mg}_{0.3}\text{Ti}_{0.7}\text{-42622}$ with $x=0.2$, which are shown in Fig. 3(a). The onset of SC at $T_c=36$ K is also corroborated by a distinct reduction in $1/T_1$. However, since its broad SC transition prevents us from deducing SC characteristics precisely, we focus only on normal-state properties of this SC compound. We remark that the T dependences of $1/T_1T$ for $x=0$ and 0.2 resemble those in non-doped (ND) and optimally electron-doped LaFeAsO -based compounds,^{11, 12, 25, 26} respectively, as compared in Figs. 3(a) and 3(b). The decrease in $1/T_1T$ upon cooling for $x=0.2$ is mostly attributed to the band structure effect,²⁷ suggesting the suppression of AFM spin fluctuations in low energies. Since the lattice parameters do not change so much in the series of $\text{Mg}_{0.5-x}\text{Ti}_{0.5+x}\text{-42622}$,⁴ probably due to the strong covalent bonding in the perovskite blocks, the SC in this compound takes place by increasing Ti^{4+} content through the substitution of Ti for Mg, namely, by doping electron carriers. This contrasts with the optimally electron-doped LaFeAsO with $T_c=28$ K in which either the F-substitution or the O-deficiency changes both the electron-doping level and the lattice parameters. In another context, note that the normal-state property for $x=0$ differs from the case of the related 42622 compound $\text{Ca}_4\text{Al}_2\text{O}_6\text{Fe}_2\text{As}_2$ (denoted as Al-42622),⁵ as shown in Fig. 3(a). The latter compound was characterized by the development of AFM spin fluctuations at low energies²⁸ in association with the nesting between the hole and electron Fermi surfaces (FSs) being quite better²⁹ owing to the lattice parameters characterized by a very short $a = 3.71$ Å, a narrow $\alpha \sim 102.1^\circ$,

and a high $h_{Pn} \sim 1.50 \text{ \AA}$.⁵ By contrast, MgTi-42622 with a nearly ideal FeAs₄-tetrahedron possesses slightly worse FS nesting properties of than that of Al-42622,³⁰ but T_c is higher than in Al-42622. This result suggests that AFM spin fluctuations are not only a unique parameter for enhancing T_c . We also note that a highest $T_c=36 \text{ K}$ in the series of Mg_{0.5-x}Ti_{0.5+x}-42622 with the optimum electron doping level at $x=0.2$ is higher than $T_c=28 \text{ K}$ in optimally doped LaFeAsO, which should be ascribed to the fact that the local tetrahedron structure of FeAs₄ is optimized. Within a spin-fluctuation mediated pairing theory on a five-orbital model, Usui *et al.* have theoretically claimed that not only the nesting of the hole and electron FSs but also the multiplicity of FSs are important to realize high- T_c SC in Fe based compounds.³⁰ According to this scenario, the higher T_c in Mg_{0.5-x}Ti_{0.5+x}-42622 can be attributed to the larger multiplicity of FSs in Mg_{0.5-x}Ti_{0.5+x}-42622 than in Al-42622, whereas the nesting property of FSs are not perfect. Further systematic studies on the relationship between the local structure and electronic state in the related 42622 compounds are desired.

In summary, the ⁷⁵As-NMR studies on Sr₄(Mg_{0.5-x}Ti_{0.5+x})₂Fe₂As₂O₆ have unraveled that the AFM stripe order takes place for $x=0$ and the SC sets in at $T_c=36 \text{ K}$ for $x=0.2$. The increase of Ti substitution from $x=0$ to 0.2 brings about the onset of SC with $T_c = 36 \text{ K}$ as a result of doping electron carriers into FeAs layers, which resembles the variation of the electronic states in the electron-doped LaFeAsO compounds through either F-substitution or O- deficiency. The phase diagram of the Fe-pnictides with thick perovskite-type blocking layers resembles those in other Fe-based superconductors which emerge in close proximity to the AFM phase by doping either electron or hole carriers. As for the SC state, the comparison with the related 42622 compound Ca₄Al₂O₆Fe₂As₂ with $T_c=27 \text{ K}$ suggests that antiferromagnetic spin fluctuations are not a unique factor for enhancing T_c . The reason why the $T_c=36 \text{ K}$ at $x=0.2$ is higher than the optimally electron-doped LaFeAsO with $T_c=28 \text{ K}$ may relate to the fact that the local tetrahedron structure of FeAs₄ is optimized for the onset of SC.

This work was supported by a Grant-in-Aid for Specially Promoted Research (20001004) and by the Global COE Program (Core Research and Engineering of Advanced Materials-Interdisciplinary Education Center for Materials Science) from the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan.

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